# THE ANALYSIS OF DIFFERENT FRACTURE TOUGHNESS MODELS FOR PVD COATINGS

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**Abstract**

The materials' resistance to brittle fracture is a very important parameter for the characterization of PVD coatings. The fracture toughness of solid materials is determined by the critical value of the stress concentration coefficient $K_{Ic}$ [1,2]. There are different models for determining the $K_{Ic}$ coefficient of thin coatings. The most often used is the method proposed by Niihara [3] and Laugier [4].

In this work, the authors present the possibility of characterization of the fracture toughness in mode I ($K_{Ic}$) for selected PVD coatings. The Young's modulus, hardness and fracture toughness of these coatings are extracted from nanoindentation experiments. The fracture toughness was evaluated using the calculation of crack length measurement, which was generated by the Vickers and Berkovich indenter. An important observation is that it is possible to use the micro indentation and nanoindentation methods for the mechanical characterization of micro-volume systems.

**Keywords:** PVD coatings, fracture toughness, Laugier model, Niihara model

## 1. INTRODUCTION

The fracture toughness in mode I ($K_{Ic}$) is determined by the material's resistance to brittle cracking. Recently much attention has been focused on a large amount of fracture toughness ($K_{Ic}$) especially for thin coatings, using the nanointendance method. The use of penetration method to study of the fracture toughness $K_{IC}$ was proposed in the 1970s by Evans and Charles [5], who observed the relation between crack lengths, which was generated in the corners of Vickers [6] indenter during the hardness test and the value of $K_{Ic}$ [7]. There are less information about using of the nanoindentation technique with Berkovich indenter dedicated to Laugier model [4], which determined the fracture toughness for thin coatings. The first work in this area were the research of Laugier [4], Dukino [8] and Ouchterlony [9], who modified the Niihara model [3]. The limited values of the $K_{Ic}$ parameter test for coatings using the nanoindentation method also result from the great difficulties in interpreting the obtained results, which is due to the significant and often deliberate heterogeneity of the coatings, including e.e. multilayer and phase differentiation. Our motivation was the lack of systematization of methodological principles in fracture toughness analysis for PVD coatings, including: the hardness range of coatings where the use of the method is justified, the possibility of comparing coatings with different parameters (e.g. hardness thickness) or the influence of the substrate on the obtained results. The mechanical characterization of PVD coatings is of great importance for the process of optimization and development of material solutions

In the article, we focused on showing the possibility of measuring the $K_{Ic}$ coefficient for thin PVD coatings with the use of analytical models by Niihara [3] and Laugier [4]. The aim of the research was to propose a method for determining the $K_{Ic}$ coefficient of thin PVD coatings using the nanoindentation method. In the article, the Laugier model was used to analyze changes in fracture toughness ($K_{Ic}$) for ceramic-based $TiB_2$ coatings doped with chromium. For comparison is shown Niihara model, which was used to analyze of $K_{Ic}$ coefficient for TiN coating deposited by an arc evaporation method. Both models give a good tool for analyzing this type of coating.

## 2. METHODOLOGY

### 2.1. Coating deposition

$TiB_2$ coatings doped with different concentrations of chromium were obtained by DC magnetron sputtering method using original magnetron systems made by Łukasiewicz Research Network - Institute for Sustainable Technology in Radom (Ł-ITeE Radom) with a Balzers pump system (Radom, Poland) with two circular magnetrons placed at an angle of 120º to each other. In the deposition process was using two targets made of $TiB_2$ and pure - Cr ( diameter d=100 mm and thickness g=7 mm) similar to the procedure presented in the paper [10]. The $TiB_2$ and Ti-B-Cr coatings were deposited in an atmosphere of pure argon (Ar 100 %). The tested coatings were deposited on the samples made of steel W320 (42,5 HRC). Before deposition, the chamber was evacuated to the pressure 4 x $10^{-3}$ mbar and substrates were heated up to 300ºC. The $TiB_2$ deposition was carried out under imput power 2000 W and power of chromium: 70 W, 100W and 165 W. The deposition time of each coating was 60 min.

For the purpose of analysis of the thickness of deposited coatings on the cracking mechanism intensity, the TiN coating was chosen. The technological process was performed by using PVD Standard 3 technical devices using the arc evaporation method with the source current I=60 A. The TiN coating were deposited on steel S600(61,5 HRC).

### 2.2. Microstructure and mechanical properties

The analysis of the microstructure was carried out using a Hitachi TM3000 Scanning Electron Microscope (SEM). Samples of W320 and S600 coated with the deposition coatings were subjected to hardness testing and Young's modulus. The results of the basic mechanical properties of the $TiB_2$ coatings and $TiB_2$ doped with chromium, including hardness and Young modulus were performed using the CSM – TTX/ NHT2 CSM – TTX/NHT2 nano-hardness tester by Anton Paar. Maximum penetration depth was determined by the thickness of 10% of the total thickness of the coating according to the procedure. For each of the tested samples were performed 15 measurements of hardness and Young's modulus. Then 10 mean values were determined from among 10 representative measurements. The procedure of investigating mechanical properties was the same for TiN coating.

### 2.3. Fracture toughness

One of the methods which are dedicated to the evaluation of fracture toughness for PVD coating is the Niihara model. Niihara model [3,11,12] is based on the Vickers indenter. Niihara formula contains the Vickers hardness (HV) and Young's modulus of the tested material. The most important relationship, which allows the selection of the model is the value of the ratio *l*/*a* (*l* - length of cracks, *a* – diagonal of the indenter), which should be in the range 0.1 <(*l*/0.5*a*)<1.5. The scheme of the cracks and method of analysis are shown in **Figure 1**. For generating visible cracks, which are necessary for fracture toughness analysis the FutureTech hardness tester was used, which is equipped with Vickers indenter. This tester allows for making measurements in the range of 0.3-30 kgf (2.9- 294.19 N). The fracture toughness was calculates in accordance with Equation 1.

$$K_{Ic} = \frac{0.035 HV\sqrt{a}}{\varphi\left[\frac{HV}{E\varphi}\right]^{0.4}\sqrt{l/a}} \quad (1)$$

where 0.035 – constant equal, *HV* –Vickers hardness [GPa], *E* – Young's modulus [GPa], $l$- the average length of cracks [μm], $a$ – the length of the half diagonal of the indentation [μm], $\varphi=3$ – factor 3.

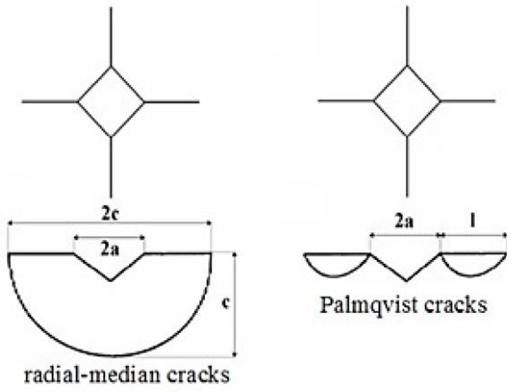


**Figure 1** The scheme of Niihara model with the using of Vickers indenter with generated cracks in the range 0.1 < (l / 0.5·*a*) < 1.5.

The Laugier [4,10,13] model is the only method for determining the $K_{Ic}$ coefficient, which uses the penetration method and a Berkovich indenter. This model is designed directly for testing smooth and thin coatings. The Laugier model takes the load applied on the indenter as well as the hardness and Young's modulus of the testing coatings (2). The schematic view of the method and analysis of the generated cracks are shown in **Figure 2**. For measurements of our samples, the nanohardness tester equipped with Berkovich indenter with the possibility of load range 5- 500 mN. The measured value was used to determine fracture toughness in accordance with Equation 2.

$$K_{Ic} = x_v \cdot (\frac{a}{l})^{\frac{1}{2}} \cdot (\frac{E}{H})^{\frac{2}{3}} \cdot \frac{P}{c^{\frac{3}{2}}} \quad (2)$$

where $x_v$ =0,016 –constant equal, $H$– hardness, $E$ – Young modulus [GPa], $P$ – applied load, $a$ – distance between corner to the center of indentation [μm], $l$ - the average crack length, $c$ – amounting $a$ and $l$.

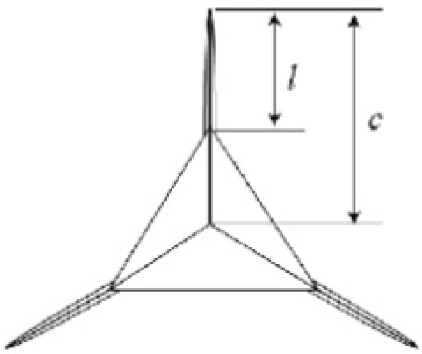


**Figure 2** Illustration of the Laugier model for determination of the $K_{Ic}$ coefficient with Berkovich indenter

## 3. RESULTS

### 3.1. Coating characterization

The analysis of the available analytical models for determination of the fracture toughness $K_{Ic}$ of PVD coatings by the penetration methods, showed that the observation of the elastic-plastic properties of the tested coatings is needed i.e. hardness and Young's modulus. For this purpose, the thickness, the hardness (H) and Young's modulus (E) were determined for all 5 PVD coatings selected for the tests. The ratios H/E and $H^3/E^2$ were calculated. The H/E determines the resistance of the coating to elastic deformation, while $H^3/E^2$ an indicator of the resistance to plastic deformation and an increase in this leads to an improvement in the load capacity. The basic mechanical properties of the presented coatings are shown in **Table 1** and **Table 2.**

### 3.2. Determination of the Fracture Toughness $K_{Ic}$

The hardness of the obtained $TiB_2$ and $TiB_2$ doped by chromium coatings is about 30 GPa. It means that this coating can be classified as a superhard coating. The $H^3/E^2$ index (plasticity deformation index) decreased with an increase in chromium concentrations.

According to the described methodology in [10,13], 20 indentations were made with selected indenter load (P) for coatings $TiB_2$, TiBCr (3%), TiBCr (6%) and TiBCr (10%). It was demonstrated, that crack initiation in the group of $TiB_2$, TiBCr (3%), TiBCr (6%) and TiBCr (10%) coatings occurs when the critical load is exceeded, which changes for various coatings and different substrate parameters. The fracture toughness test was performed by measuring the length of cracks in the corners of the indentations made with a Berkovich indenter with different loads in the range P = 200 - 500 mN based on the Laugier formula [4,10]. The average values of *l* and *a* were determined for each indentation for the whole series 20 indentations The results of the fracture toughness tests are presented in **Table 1** as a parameter $K_{Ic}$. The analysis carried out on the Laugier model presented that doping of chromium significantly influences the increase of fracture toughness value for coatings based on $TiB_2$. The view of the cracks obtained at the load P = 400 mN and the method of measuring the parameters of the generated cracks for TiBCr (3%) coatings is shown in **Figure 3**. The authors observed that values *a* are in the range 3.70 - 7.01 μm, values *l* are in the range 0.71 - 3.07 μm and the values $K_{Ic}$ are in the range 1.51 - 6.23 MPa*$m^{1/2}$.

**Table 1** The characteristic parameters of mechanical properties: thickness,load, hardness, Young's modulus and H/E and $H^3/E^2$ for $TiB_2$ and Ti-B-Cr coatings for calculation of the fracture toughness by the Laugier model.

| Coating | Thickness (μm) | Load P (mN) | Hardness H (GPa) | Young's modulus E (GPa) | H/E | $H^3/E^2$ | a (um) | l (um) | $K_{Ic}$ (MPa*m1/2) |
|---|---|---|---|---|---|---|---|---|---|
| $TiB_2$ | 1.60 | 300 | 34.5 | 410 | 0.084 | 0.244 | 3.70 | 1.73 | 2.84 |
| TiBCr(3%) | 1.73 | 400 | 33.3 | 393 | 0.085 | 0.239 | 6.60 | 3.04 | 1.60 |
| TiBCr(6%) | 1.73 | 350 | 32.5 | 388 | 0.084 | 0.228 | 5.42 | 3.07 | 1.51 |
| TiBCr(10%) | 1.79 | 500 | 30.3 | 383 | 0.079 | 0.190 | 7.01 | 0.71 | 6.23 |

The second step of our methodology work was to find the model, which can be dedicated for TiN coatings deposited by arc evaporation. After measurement of hardness and Young's modulus of our samples deposited by arc evaporation, we choose the Niihara model [3,11]. According to the adopted methodology, metal nitride TiN coating was selected for tests deposited by the low-pressure electric arc evaporation method with a thickness of $g_{TiN}$= 3.10 μm. The indentations were made in laboratory conditions using a FutureTech hardness tester, with the following loads: 0.5 kgf (4.9N), 1 kgf (9.8N), 3kgf (29.4N), 5kgf (49 N) and 10 kgf (98 N). For each coating, there are made some indentations with selected P, which is higher like in TiB2 coatings and better to show in kgf. The observations and measurements of fracture parameters were made using the Hitachi TM3000 Scanning Electron Microscope (SEM). The value of Young's modulus was determined by the penetration method using the CSM – TTX/ NHT2 CSM – TTX/NHT2 nano-hardness tester by Anton Paar, while the HV hardness value was the value determined during the making of the impression. In addition, the influence of coating thickness was assessed in the process of determining the $K_{Ic}$ coefficient of the tested coatings. After the first measurements series, which was performed in the entire load range, the coatings were polished for reducing their thickness by about 0.5 μm. The example of Vickers indentations for thickness 0.81 um were shown in **Figure 3b**. An important observation is, that the $H^3/E^2$ index increased with decreasing of the thickness. The average values of *a* and *l* are increasing when the thickness of coatings are decreasing.

The results of measurements of fracture toughness coefficient for TiN coatings show that the values $K_{Ic}$ are in the range 10.00 -13.00 MPa*m$^{1/2}$ for thickness coating of 3.10 um and in the range 6.23-14.18 MPa*m$^{1/2}$ for thickness coating 0.81 um (**Table 2**).

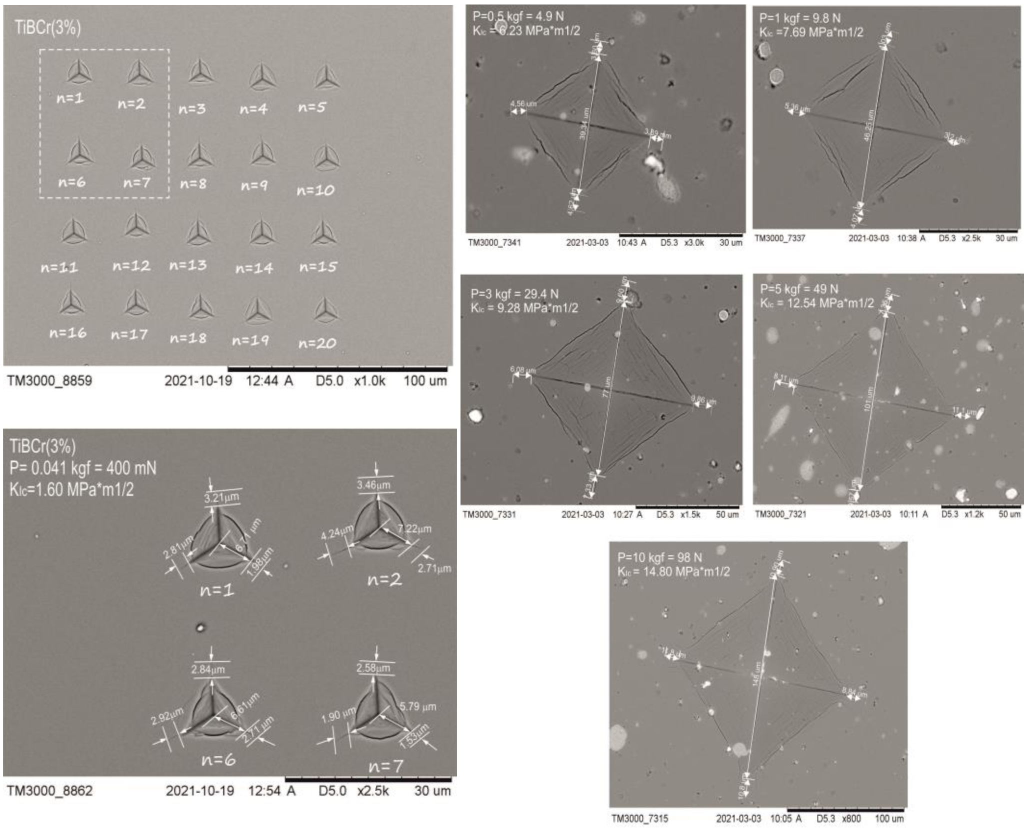


**Figure 3** The example SEM images of groups indentation used for fracture toughness analysis ($K_{Ic}$) for TiBCr(3%) coatings a) series of 20 indentations with a Berkovich indenter at a load of P = 400 mN and dimensional analysis of crack lengths for different indentations where n=1,2,6,7. b) The example SEM images of a group of indentations made with the Vickers indenter in a coating with a thickness of $g_{TiN}$ =0.81 μm at different indenter loads (0.5 kgf, 1 kgf, 3 kgf,5 kgf and 10 kgf).

**Table 2** The characteristic parameters of mechanical properties: thickness, load, hardness, Young's modulus and H/E and $H^3/E^2$ for TiN coatings for calculation of the fracture toughness by the Niihara model.

| Coating | Thickness (μm) | Load P (kgf) | Hardness HV (GPa) | Young's modulus E (GPa) | H/E | $H^3/E^2$ | a (um) | l (um) | $K_{Ic}$ (MPa*m$^{1/2}$) |
|---|---|---|---|---|---|---|---|---|---|
| TiN | 3.10 | 3 | 25.7 | 388 | 0.066 | 0.113 | 38.2 | 8 | 10.16 |
| | 3.10 | 5 | 25.7 | 388 | 0.066 | 0.113 | 55 | 10 | 12.00 |
| | 3.10 | 10 | 25.7 | 388 | 0.066 | 0.113 | 71.66 | 15 | 13.00 |
| | 0.81 | 0.5 | 20.4 | 353 | 0.060 | 0.070 | 16 | 3 | 6.23 |
| | 0.81 | 1 | 20.4 | 353 | 0.060 | 0.070 | 22 | 4 | 7.69 |
| | 0.81 | 3 | 20.4 | 353 | 0.060 | 0.070 | 39 | 7 | 9.28 |
| | 0.81 | 5 | 20.4 | 353 | 0.060 | 0.070 | 51 | 8 | 12.54 |
| | 0.81 | 10 | 20.4 | 353 | 0.060 | 0.070 | 73 | 11 | 14.80 |

## 4. CONCLUSION

In the article, the authors presented the basic information about using Niihara and Laugier models for determining $K_{Ic}$ coefficient of different PVD coatings. The very important observation is that values of $K_{Ic}$ are increasing with the concentration of chromium for coating based on $TiB_2$. The highest value of $K_{Ic}$ was calculated for TiBCr(10%) ($K_{Ic}$=6.23 MPa*$m^{1/2}$) deposited on steel W320. For calculation of $K_{Ic}$ for this type of coatings Laugier model was used. Another area of analysis was investigated of TiN coating with thickness around of 3.10 μm deposited by arc evaporation method. In this particular case, the authors decided to used Niihara model with Vickers intender for determining $K_{Ic}$ values of these coatings. Our investigation show that the values fracture toughness are increasing with decreasing of the coating thickness. Our short analysis, which the authors presented in the paper show that it is possible to use Niihara and Laugier model for the evaluation of resistance to the brittle cracking of selected PVD coatings.

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